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Analysis of Zirconia Powder for Thermal Spray: Reference Material for Particle Size Distribution Measurement

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Abstract

The thermal spray industry identified the need for repeatable and reproducible feedstock powder characterization methods, especially particle size distribution (PSD), for cost effective manufacturing of thermal barrier coatings. The PSD measurement by a laser light scattering method was identified as the technique most widely used in the industry. This technique offers high resolution, rapid measurements and ease of use.

A round robin study by nine laboratories using different models of a commercial light scattering instrument has been completed as the first step towards the development of a Standard Reference Material (SRM) for the calibration of light scattering instrument. Other measurement techniques were also employed for additional comparison. The PSD measurements employing light scattering techniques evidenced some method dependence, despite the use of identical sample preparation procedures. The round robin results will serve as reference values for the development of the SRM.

THERMAL SPRAY DEPOSITION of ceramic coatings is a significant materials processing technology. Yttria stabilized zirconia (YSZ) is one of the powders used widely with the main application being thermal barrier coatings for aircraft engines and aerospace applications and to a lesser degree in diesel engines. Major concerns in both applications are the yield of coating from a given quantity of powder and the reproducibility of coating properties.

At the 1992 NIST/Industry Workshop on Thermal Spray (1), powder characterization and powder quality control were identified as issues which have a major impact on the manufacture of cost-effective thermal barrier coatings. The development of repeatable and reproducible measurement methods particularly for particle size distribution (PSD) requires the use of standard reference materials (SRM). Therefore, the development of an SRM for PSD of a YSZ powder was initiated.

The first requirement for the production of this SRM was the identification of a YSZ powder with: a) a well-defined monomodal PSD, b) spheroidal particles, c) little agglomeration and d) particles of high mechanical strength and low friability. Then, a procedure for the PSD measurement had to be specified.

To assess both the material and the procedure, a round robin study was conducted. Since all of the participants of this study used Microtrac^{R} Instrument* for PSD analyses, the results pertain only to this specific class of instruments. The round robin study was conducted among participants which included powder producers and users, as well as instrument and equipment manufacturers. The data were evaluated statistically at NIST.

Materials and PSD Measurement Techniques

YSZ Powder. YSZ powders are manufactured by many processes such as: sol-gel precipitated and sintered; spray dried and sintered or plasma densified; fused and crushed or agglomerated and sintered. Powder samples of each of the above manufacturing processes were obtained for evaluation.

*Certain trade names and compasny products are mentioned in the text or identified in illustrations to specify the experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply the products are necessarily the best available for the purpose.

After a thorough study of thirteen powders produced by the processes listed above, a candidate powder which was produced by a "spray dried and sintered" process by Metallurgical Technologies Inc. (METEC), Pearland, Texas was chosen. A specific lot was procured after evaluating three production batches of such powder for their physical integrity and powder morphology.

Particle shape and degree of agglomeration determined by scanning electron microscopy (SEM) imaging at different magnifications are shown in Figure 1. The SEM micrographs show the powder to be comprised of spheroidal particles with a low degree of agglomeration. The chemical composition of powder provided by the manufacturer is listed in Table 1.

Table 1: Major Chemical Components in Yttria Stabilized Zirconia (provided by Metallurgical Technologies)

	Mass Fraction,%
Yttrium Oxide	7.33
Hafnium Oxide	1.39
Silicon Oxide	0.13
Titanium Oxide	0.08
Aluminum Oxide	0.02
Calcium Oxide	0.01
Ferric Oxide	0.02
Magnesium Oxide	0.01
Uranium + Thorium Oxide	0.01
Zirconium Oxide	90.0*

*This mass was calculated by assuming the balance was zirconium oxide.

Low friability of the powder was demonstrated by comparing the PSD of the powder before and after sonicating in a water-based slurry with a high wattage sonic horn. Figure 2 shows the PSD of powder before and after sonication as measured by a Horiba LA 900 light scattering instrument. Only a small change in PSD was detected even after this high power of sonication (45 W for 2 min). This low friability ensured us that the particles breakage will be insignificant during transportation and use.

Representative samples were packaged with the use of a spinning riffler. Each sample contained 10 g powder. A randomized set of 100 samples were selected for homogeneity testing and SRM certification.

PSD Measurement Techniques. Numerous PSD measurement techniques are described and compared in technical literature (2-7). The following three techniques were investigated: a) laser light scattering, b) SEM imaging analysis, and c) sieving.

Laser Light Scattering. Application of the laser light scattering technique to measure PSD has gained wide acceptance due to its speed of measurement, high repeatability and high resolution. There are several instruments on the market. The following three instruments have been included in this study: (I) Microtrac^R (Leeds & Northrup Co., St. Petersburg, FL) (ii) Horiba LA 900 (Horiba,Irvine, CA) and (iii) Coulter LS-130 (Coulter Corp., Hialeah, FL). Each instrument derives PSD data from laser diffraction data using proprietary algorithms.

The Microtrac^R instrument has been the dominant instrument used in the thermal spray community because of its early availability to the industry. The Microtrac^R instrument also offered many advantages over the previously used sieving measurement method such as high resolution, reproducibility and ease of use. A comparison study of the Microtrac^R instrument and sieving was reported (8). In the above comparison study, the measured PSDs were finer for sieving than for Microtrac^R measurements although the cause was not given.

Nine laboratories representing the powder producers, powder users, plasma gun and PSD instrument manufacturers having Microtrac^R instruments participated in this round robin study to determine (assign) the PSD reference values for this SRM. Each round-robin participant received four samples for analysis using their model of the Microtrac^R instrument. The same sample preparation procedure specified by NIST was used in both homogeneity testing and analyses.

SEM Image Analysis. The direct imaging of zirconia particles by scanning electron microscopy provides a method by which absolute size measurements may be made. The SEM can provide high resolution (.01μm) length measurements with magnifications calibrated directly with NIST length standards. The large depth of field intrinsic to electron beam instruments provides a clear advantage over light optical imaging for analysis of spherical particles such as these spray dried materials. The nature of this imaging procedure is such that the projected area of each particle must be individually measured. The statistical requirements for measurement of the particle size distribution dictates a preparation procedure for the microscopy samples designed to achieve a balanced sampling of the different size fractions, and a balanced statistical measure of each size fraction. The stringent sample preparation requirements and the necessity of measuring thousands of individual particles requires considerable time and effort. SEM-image analysis is therefore used mainly for calibration tests with more rapid but less absolute techniques being used for PSD measurements on a routine basis. The SEM measurements will be used in the certification of this zirconia material as an SRM.

Sieving. The bulk separation of powder materials by sieving through wire mesh screens has long been a standard procedure. This technique is restricted in size resolution by the availability of screens within the limited set of standard mesh sizes. The repeatability of this technique depends

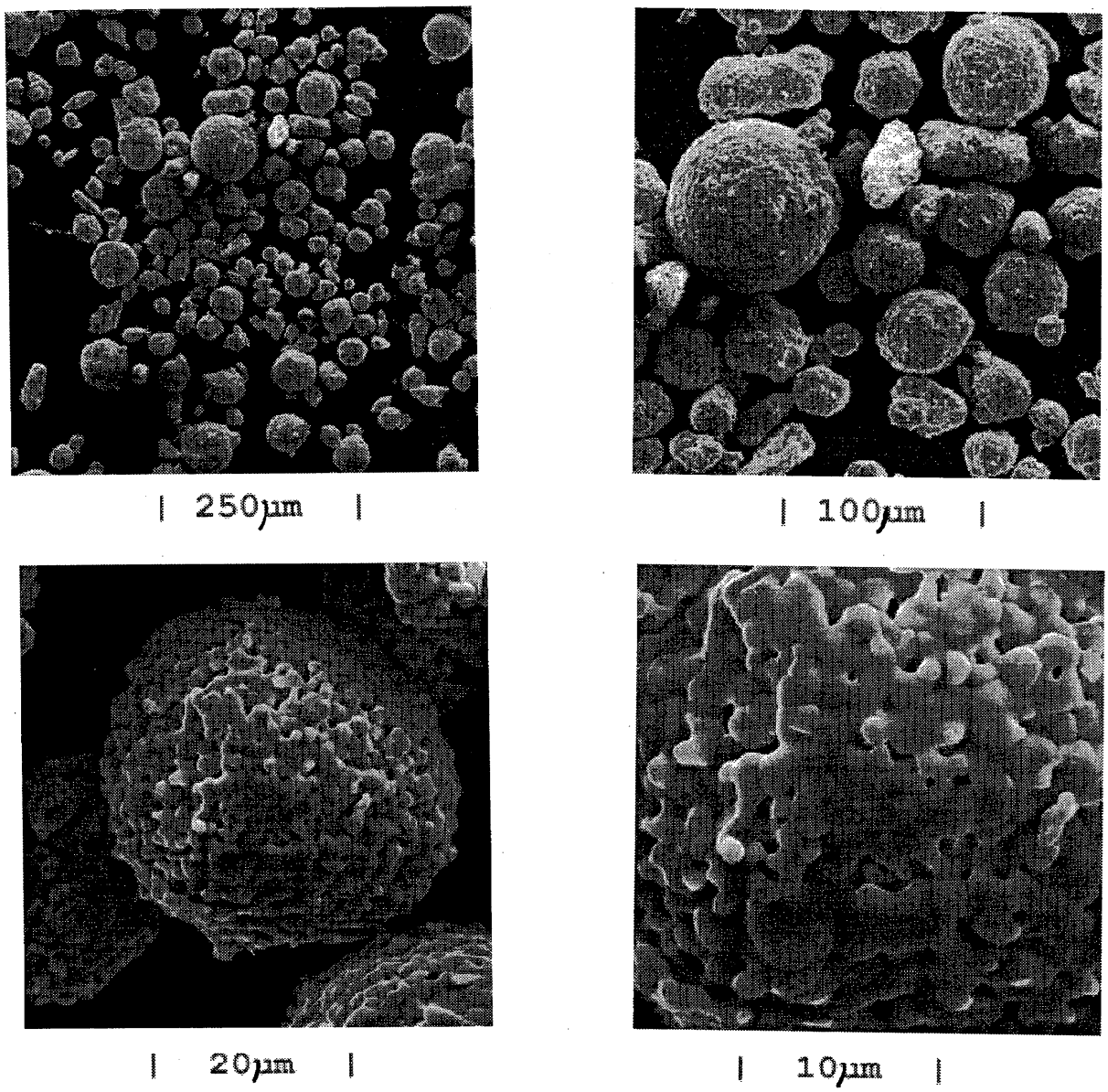

Figure 1. SEM Micrographs of yttria stabilized zirconia powder at different magnifications to illustrate powder and particle morphology.

greatly on the tedious and time consuming procedure of weighing and cleaning of each sieve after every test. This method will also be used as part of the SRM certification.

Experimental Procedure

Sample Preparation. Uniform and consistent test sample preparation techniques were necessary for the evaluation of PSD regardless of the type of light scattering instrument. Each sample vial contains 10 g powder; sufficient for one PSD analysis by older Microtrac^R instruments. For newer Microtrac^R instruments, such as model X-100, a smaller sample size is used and a micro-riffler is recommended for splitting the 10 g sample into subsamples of the desired mass. Distilled water with pH adjusted to 9.5 is used for both the preparation of ceramic powder slurry and PSD measurements. Dilute sodium hydroxide (0.1 mol/L) may be used to adjust the pH of distilled water. Dispersion of the powder is accomplished by making a paste of the powder by adding separately prepared 4% (by mass) sodium pyrophosphate solution at the ratio of 0.5 cm^3 solution per gram of powder. The paste is transferred quantitatively (totally) into the measuring cell containing pH-adjusted 9.5 distilled water. The transfer of the paste to the cell can be achieved by flushing the container with additional pH-adjusted distilled water.

The size measurement is carried out by following the instrument manufacturer's procedure for instrument operation, and determination of PSD.

Homogeneity Testing. To measure the PSD data variability among the sample vials prepared for SRM distribution, a homogeneity study was completed before starting the certification study. A random set of samples was chosen and their PSD measurements were completed in duplicate and in random order. The homogeneity study indicated no significant variance among the samples prepared. Therefore, the homogeneity study data were also used in the certification analyses.

Both NIST and Leeds & Northrup Co. (L&N) participated in the homogeneity study. One random set of 15 samples was selected for analyses at NIST using a Horiba LA 900 instrument and another set of 10 samples was sent to L&N for both homogeneity testing and certification analyses using the Model X-100 Microtrac^R instrument.

Horiba LA-900 at NIST. The PSD of 15 samples chosen for this homogeneity study was determined in triplicate and in random order, using the Horiba LA-900. The same sample preparation procedure specified above was used. The size measurement was carried out by following the Horiba procedure for instrument operation and determination of PSD. The following instrument parameters were used: (a) Sample size for each analysis was 0.2 g, (b) relative refractive index (RRI) was 1.8 with respect to that of water (refractive index of Zirconia powder was set at 2.4 and refractive index of water was 1.33). This optimum RRI for Horiba Instrument was derived from the following study. The procedure for this empirical determination of optimum RRI was described by Hayakawa et al (9). Figure 3 shows the change of particle size at d_{50} as a function of RRI from 1.5 to 2.7. The particle size data were calculated on the basis of volume with a normal distribution. The particle size at d_{50} changed from 60.2 μm to 61.2 μm which was well within experimental uncertainty** (±0.8 μm) and a negligible effect on PSD measurement. However, d_{50} is the maximum at RRI=1.8.

Although the Horiba LA-900 instrument produces a continuous plot of mass fraction percentage finer than a given diameter, five cumulative percentiles (10%, 25%, 50%, 75% and 90%) were selected as a representative data set consistent with the industrial practice.

Microtrac^R X-100 at L&N. The PSD of 10 samples were determined in duplicate using the Microtrac^R X-100 at the L&N laboratory. The same sample preparation procedure described above was used. Particle refractive index was at 2.2 and the fluid (water) refractive index was at 1.33 for the later models of Microtrac Instruments.

PSD Data from other Procedures. PSD measurements, not homogeneity analyses, were conducted with sieving, SEM, and Coulter LS-130.

Sieving Analysis. Seven samples were analyzed by sieving using the following mesh screens: 120 (125 μm), 170 (90 μm), 200 (75 μm), 230 (63 μm), 270 (53 μm), 325 (43 μm) and 400 (38 μm). A sonic sifter by ATM Corp. (Milwaukee, WI) was used in this analysis at a medium sonic amplitude and in the pulse/shift mode. Each sample charge was approximately 10 g and sifting was conducted for 10 min. The mass of each sieve fraction is measured with a resolution of 0.001 g.

SEM Image Analysis. SEM analysis was carried out on two samples of 10 g vials. Sample preparation for microscopy included both a reduction in the mass of powder and a separation into size fractions. The size fractionation was accomplished by sieving with a Sonic Sifter using U.S. Standard Series sieves numbers 120 (125μm), 140(106μm), 170(90μm), 200(75μm), 230(63μm), 270(53μm), 325(45μm) and 400(38μm). Subsamples from each of the sieve splits were then produced by successive division using a spinning riffler. Each subsample was mixed with a 4% sodium pyrophosphate solution, suspended in water with pH adjusted to 9.5 using sodium hydroxide, and filtered onto a nylon screen. The particles were removed from the screen by pressing an SEM stub, covered with adhesive, to the filter. Examination of the filter under an optical microscope confirmed the removal of all particles from the selected section. The particles were then coated with a

** All uncertainties have a joint level of confidence of 95%.

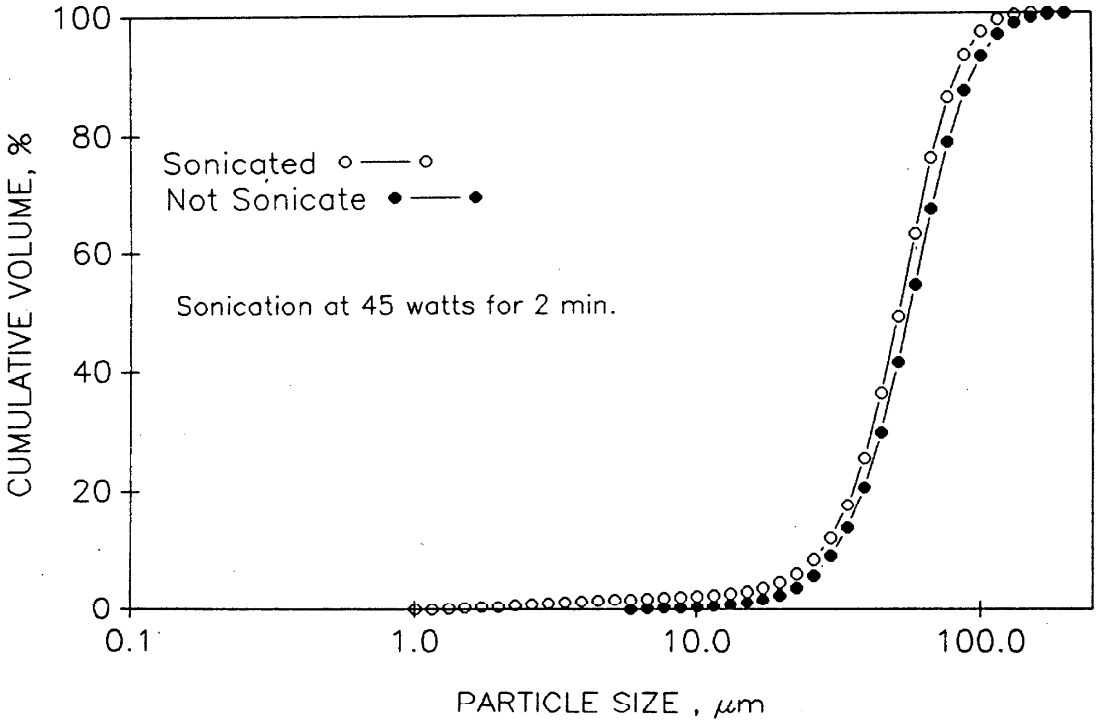

Figure 2. Particle friability of yttria stabilized zirconia powder by sonication

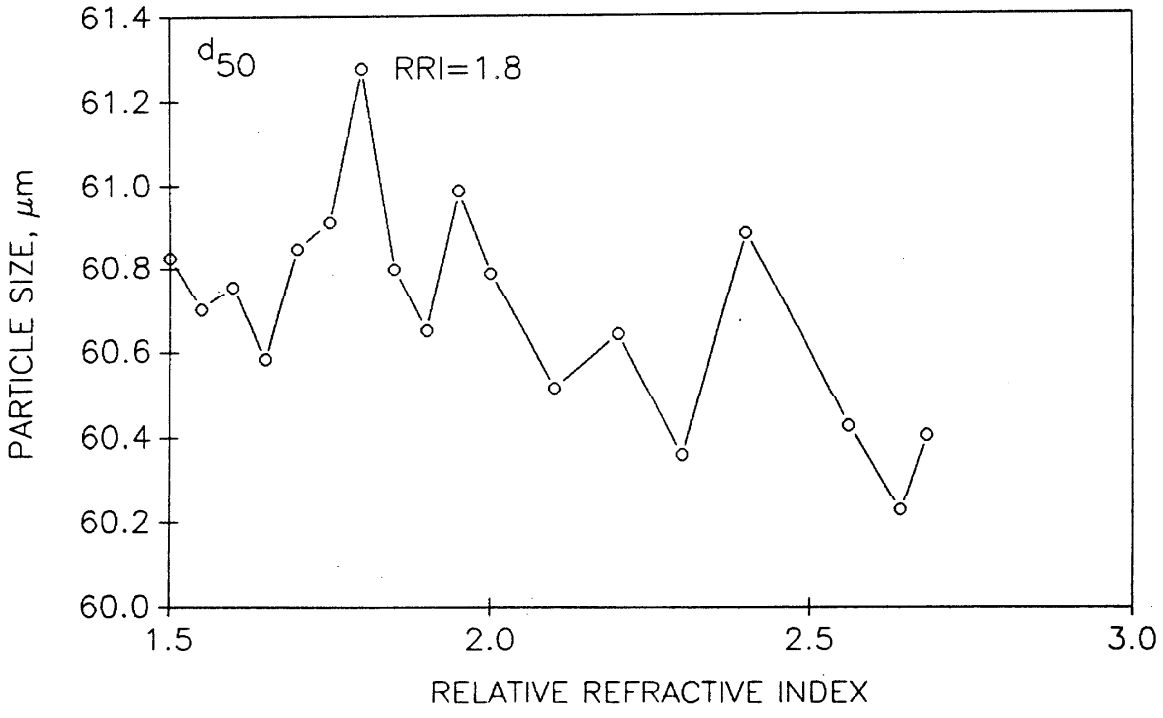

Figure 3. Effect of RI on particle size by Horiba LA 900

gold-palladium film for electrical conductivity.

SEM images were acquired for each of the nine sieve fractions. The backscattered electron images of the particles were acquired as grey-scale image files into a Apple Power PC 8100 computer via a 4Pi digital interface. The 1024 by 1024 pixel images were analyzed using the program NIH Image to obtain the projected area of each zirconia particle. These areas were converted to particle volumes and particle diameter based on the assumption of spherical particle shape. The pixel to length conversion is calibrated by collecting digital images of a calibrated standard (NIST SRM 2090) and of a micrometer slide measured at NIST using laser interferometry. The two calibration standards agree to within a length uncertainty of 1%. Particle size distributions describing the percentage of powder volume represented by particles with diameters less than a given length are calculated using the weighting factors obtained from sieving results. A PC based spreadsheet program was used to calculate particle frequency and cumulative mass distributions. The mass fraction distributions are identical to the volume fraction distributions. This is independent of particle density with the assumption that all particles have the same density.

Analysis by Light Scattering Instruments, Coulter LS 130. Samples with the same preparation as described above, were also analyzed by the Coulter LS 130 instrument at the Coulter Laboratory, Hialeah, FL. The RRI value used in this experiment was 2.0.

Reference Analyses

Nine laboratories participated in this round robin study to assign PSD reference values by the Microtrac^R technique. Each participant, except L&N, received four randomly selected samples together with the instructions for sample preparation procedures. Each participant reported the data to NIST for statistical analysis.

Technical Data on the SRM Powder

Additional technical data on the powder is included in the certificate (Table 2) for users of this SRM. These are not certified values and were determined at NIST using the following methods.

(i) Specific Gravity was determined by helium pycnometry (Micromeritics' Autopycnometer 1320).

(ii) Tap Density was determined using ASTM B527-93 method with a Dual Autotap, Quanta Chrome Corp. Boynton Beach, FL.

(iii) Both Hall Apparent Density and Hall Flow Rate were determined using ASTM B212-89 and B213-90 methods respectively.

(iv) Specific Surface Area by Nitrogen Gas Adsorption Method (Quanta Autosorb-1, Quanta Chrome Corp. Boynton Beach, FL) was determined using the method specified by the manufacturer.

(v) Powder morphology micrographs (Figure 1) were obtained using AMRAY 1830 Scanning Electron Microscope.

Table 2: Additional Technical Data on the Zirconia Powder

Specific Gravity by He pycnometer: 5.86 $\pm$ 0.01 g/cm^3
Tap Density: 2.47 $\pm$ 0.02 g/cm^3
Hall Apparent Density: 1.82 $\pm$ 0.02 g/cm^3
Hall Flow Rate: No Flow
Specific Surface Area by BET Method: 0.40 $\pm$ 0.01 m^2/g

Results

Homogeneity Results. Statistical analyses of both sets of data by Microtrac^R and Horiba LA-900 showed no evidence of inhomogeneity. The mean values of d_{10}, d_{25}, d_{50}, d_{75} and d_{90} of the samples by both Horiba LA-900 and Microtrac^R are listed in Table 3 and the variability of the PSD measurements by Horiba LA-900 is well within experimental uncertainty at a confidence level of 95% as shown in Figure 4. However, there is a substantial bias between the data determined by the Horiba LA-900 and Microtrac^R X-100 as illustrated in Figure 5 together with SEM, sieving and Coulter results.

SRM Certification

Microtrac. Microtrac reference data were consistent with the preliminary SEM and sieving data from 30 μm to 60 μm, and diverge somewhat at higher particle sizes (Figure 5). Since the Microtrac data are method dependent, the resulting reference values were statistically analyzed separately from the certified values and are listed separately on the certificate for use in the calibration of Microtrac instruments. Five cumulative percentiles were selected as a representative data set for assigning reference values and weighed averages of each participant's data are listed on Table 4 and plotted in Figure 6. Table 4 lists the averages of measurements made at each of the nine participating laboratories.

The reference values in Table 5 are weighted averages of the laboratory means which included 42 individual percentile measurements. Each reference value is the mean of measurements made in nine laboratories. The expanded uncertainties in Table 5, computed

Table 3: Homogeneity Study by Horiba LA-900 and Microtrac^R, Mean Values (μm) and standard deviations

	d_{10}	d_{25}	d_{50}	d_{75}	d_{90}
Horiba LA-900	29.9±0.7	43.8±0.9	62.0±0.9	83.9±1.2	100.3±1.8
Microtrac^R	24.5±0.2	36.3±0.4	51.9±0.6	69.1±0.9	89.7±1.6

Table 4: Reference Data from Round Robin Study on Zirconia Powder

	Weighted Average Particle Size (μm) and standard deviations				
Laboratory/ Microtrac^R Model	d_{90}*	d_{75}	d_{50}	d_{25}	d_{10}
"A"/ X-100	89.7 ±1.6	69.1 ±0.9	51.9 ±0.6	36.3 ±0.4	24.5 ±0.2
"B"/ X-100	89.6 ±0.7	72.8 ±0.4	55.4 ±0.5	37.3 ±1.9	25.8 ±0.5
"C"/ #7997	97.4 ±0.9	72.5 ±0.4	52.6 ±0.3	35.1 ±0.2	23.5 ±0.2
"D"/ X-100	94.4 ±4.0	73.8 ±3.3	53.6 ±2.9	36.8 ±2.4	24.5 ±1.6
"E"/9220-4	91.8 ±0.6	69.6 ±0.3	51.1 ±0.2	35.9 ±0.1	24.9 ±0.1
"F"/7995-12	101.9 ±0.5	78.4 ±1.0	55.5 ±0.4	37.6 ±0.5	25.1 ±0.3
"G"/7995-10	96.6 ±5.6	75.9 ±2.9	54.2 ±2.4	36.3 ±1.8	23.7 ±1.7
"H"/158704-1	98.9 ±2.1	75.9 ±0.7	53.5 ±0.3	35.4 ±0.2	23.4 ±0.1
"I"/SRA#7995-11	100.0 ±1.9	74.6 ±1.7	50.6 ±0.8	34.9 ±0.6	24.1 ±0.4
Maximum	101.9	78.4	55.5	37.6	25.8
Minimum	89.6	69.1	50.6	34.9	23.4

* For example, d_{90}, is the particle size in micrometers through which 90% of mass passes; i.e., 90% of the mass is finer than 89.7 μm

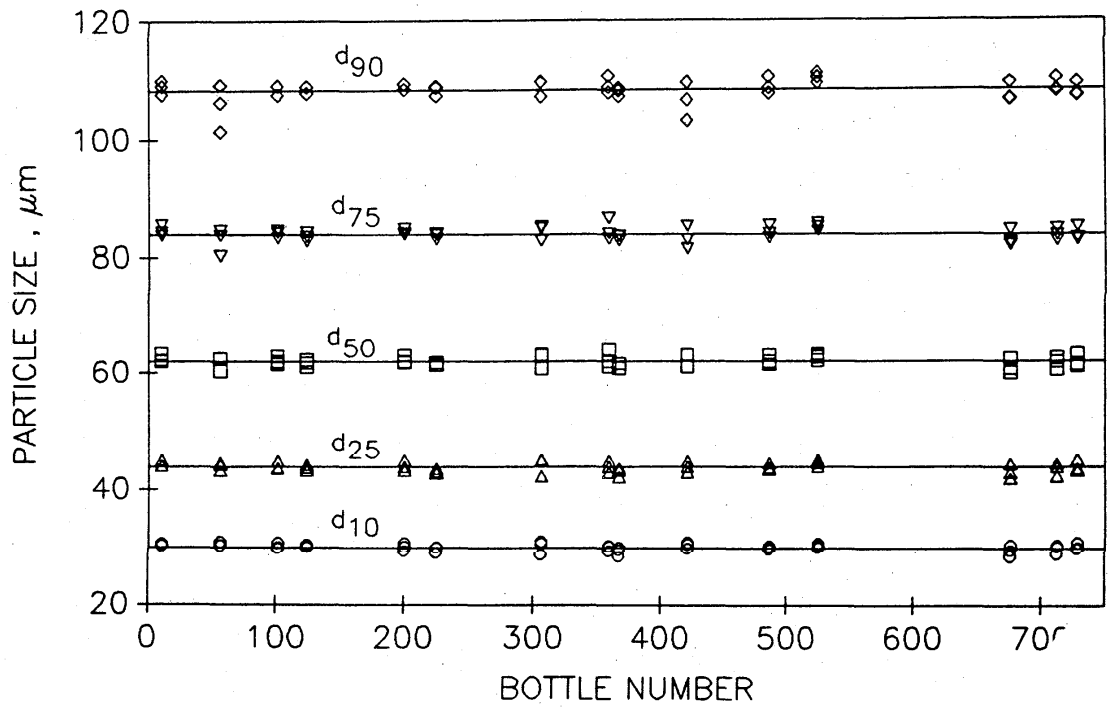

Figure 4. Measure of variability between bottles by Horiba LA 900

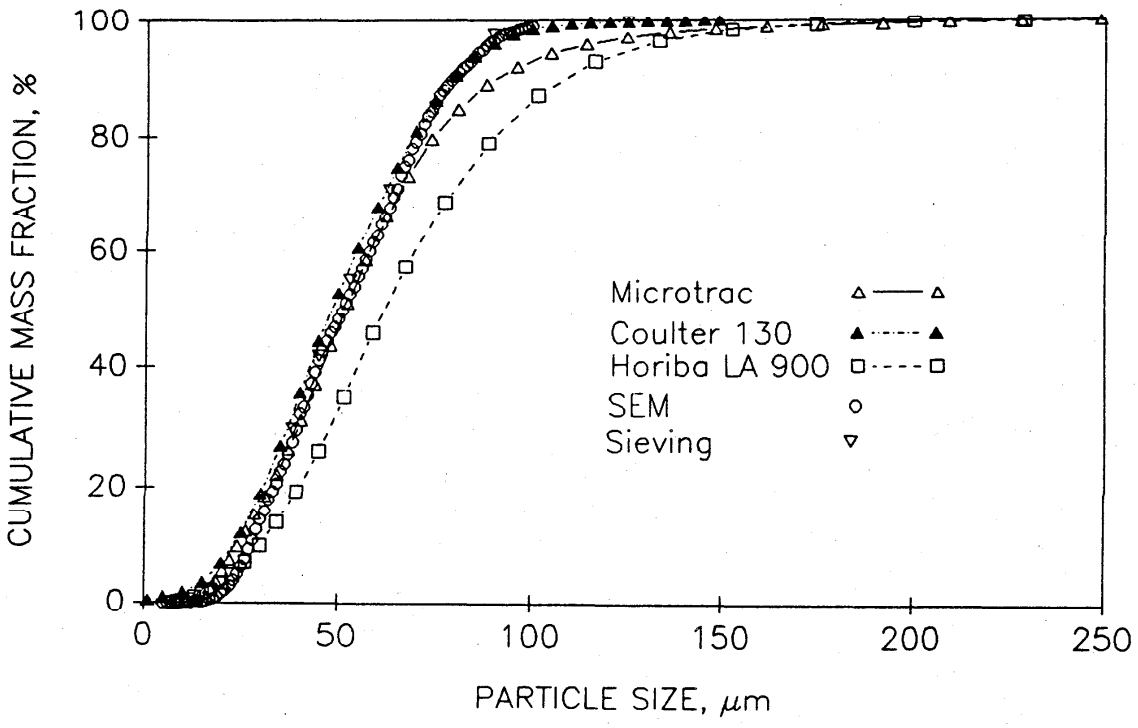

Figure 5. PSD of zirconia powder by different techniques

according to the CIPM method (10), include between and within -laboratory uncertainty and have a joint level of confidence of 95%. The five pairs of reference values and expanded uncertainties define a range within which the true percentiles of the distribution are expected to lie with approximately 95% confidence for all five percentiles considered together.

Sieving. The preliminary sieving results are listed in Table 6. The mass fraction of particles with diameters greater than 125 μm were determined by sieving to be less than 0.1%, compared to 3% by Microtrac^R model X-100 instrument. To verify that the sonic sifter did not break large particles during the sifting, a study by hand sifting was carried out. Eight-inch sieves of 100, 120, 140 and 170 mesh screens were stacked to hand screen a 56 g sample gently. The mass fraction of particles greater than 125 μm in diameter was 0.05±0.01% which was far less than the 3% measured by Microtrac^R instruments. Since the gentle sieving didn't produce a large amount of particles greater than 125 μm, the observed difference was not due to the breaking of large aggregates during sonic sieving.

Table 5: Reference Particle Size Distribution Data for all Round Robin Measure

Cumulative Mass (Percentile)	Reference Value (μm)	Uncertainty [1] (μm)
10	24.3	0.9
25	36.1	1.0
50	53.1	1.9
75	73.6	3.4
90	95.6	5.0

[1]The uncertainties computed according to the CIPM method (10), include between and within laboratory uncertainty and have a joint level of confidence of 95%.

Table 6: Preliminary Sieving Data* Summary

Screen Opening μm (# Mesh)	Cumulative Mass Fraction %
125 (120)	99.93 ± 0.18**
90 (170)	97.53 ± 2.02
75 (200)	85.13 ± 3.96
63 (230)	71.06 ± 3.48
53 (270)	55.14 ± 3.91
43 (325)	41.89 ± 4.46
38 (400)	29.87 ± 4.14

* Sieving data listed above are not certified
**99.93% mass fraction of the powder passed through 120 screen

SEM. The preliminary SEM analysis results are listed in Table 7 and shown in Figure 5. The PSD distribution by SEM imaging agreed with Microtrac data for particles up to 60 μm. SEM analysis found few particles greater than 100 μm. This is in agreement with sieving data.

Table 7: PSD Data on YSZ Powder By Other Techniques, (μm)

	SEM*	Horiba LA 900	Coulter 130
d_{10}	27±1	29.8±1.4	25.5±0.5
d_{25}	36±1	44.4±2.5	33.5±0.5
d_{50}	51±1	62.9±2.4	54.0±0.5
d_{75}	68±1	84.1±2.2	75.0±0.5
d_{90}	81±1	106.9±5.5	89.0±0.5

* SEM data listed in Table 7 are preliminary data and are not certified values

Other Laser Light Scattering Techniques

Horiba LA 900. The lack of agreement among SEM, Microtrac^R and Horiba data are illustrated in Figure 5. The reported particle size values at all "d" values were higher.

The correlation plot between the reference "d" values from Microtrac and the "d" values from Horiba LA 900 data is shown in Figure 7 with a Correlation Coefficient of 0.99 at the first order of regression.

Coulter LS 130. The Coulter LS-130 analyses results are listed in Table 7 and shown in Figure 5. There is good agreement between results of SEM analysis and Coulter LS 130 analysis. Coulter LS 130 data also show the absence of large particles.

Discussion

PSD measurements of powder in the range of 10 μm to 150 μm by laser light scattering technique are widely used, but data from different instruments may not be in good agreement. With a consistent sample preparation method and proper adjustment of instrument parameters, the PSD data by most techniques can come to an agreement as shown in Figure 5. Although the data did not come into full agreement, a good correlation between each technique can be established as shown in Figure 7 for the case of Horiba LA 900.

Conclusion

1. PSD of the thermal barrier zirconia powder was determined by different measurement techniques.

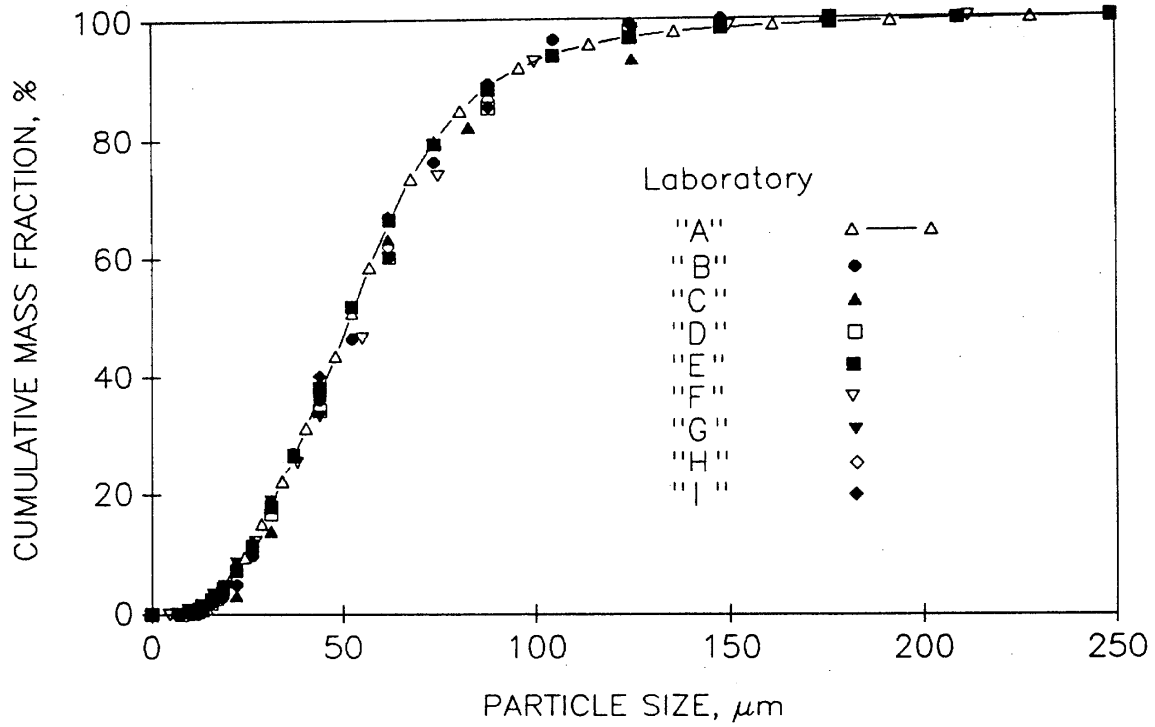

Figure 6. PSD of zirconia powder by different laboratories using Microtrac[R] instrument of different models

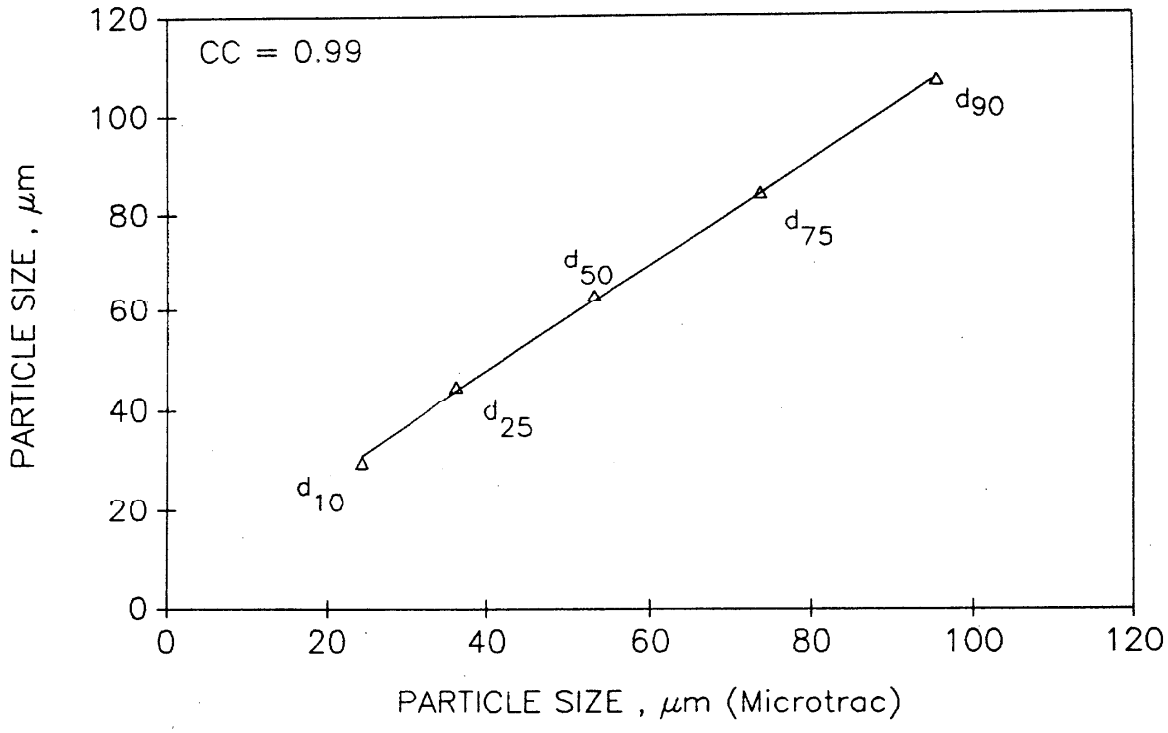

Figure 7. Correlation of PSD by Microtrac[R] and Horiba LA 900

2. Sample preparation procedure had to be controlled and a proper surfactant was used to disperse aggregates.

3. For the light scattering method, the computation format of each instrument is different and proprietary. Each laser light scattering technique was method dependent, yielding PSD as a relative value. An SRM would play an important role in calibrating instruments.

4. A round robin study on the PSD of the thermal barrier zirconia powder has been successfully completed on different models of a commercial instrument. This showed that the sample preparation procedure is critical during PSD measurements.

5. The round robin results will be used as reference values towards the development of a standard reference material.

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